

## REMARKS

In response to the above-identified Final Office Action (“Action”), Applicants traverse the Examiner’s rejection to the claims and seek reconsideration thereof. Claims 1-36 are pending in the present application. Claims 1-36 are rejected. In this response, claim 1 is amended, no claims are cancelled and no claims are added.

### **I. Claim Amendments**

Applicants respectfully submit herewith amendments to claim 1. Claim 1 is amended to recite the elements of “the light-transmitting gas selective permeable film comprising a thickness of 0.05  $\mu$ m to 1  $\mu$ m.” Support for the amendments to claim 1 may be found, for example, on page 17, lines 24 and 25 of the Application.

Applicants respectfully submit the amendments do not add new matter and are supported by the specification. Accordingly, Applicants respectfully request consideration and entry of the amendments to claim 1.

### **II. Claim Rejections – 35 U.S.C. §103**

**A.** In the outstanding Action, claims 1-3, 5 and 13-15 are rejected under 35 U.S.C. §103(a) as being unpatentable over Japanese Publication No. 2001-343380 issued to Omatsu (“Omatsu”) in view of U.S. Patent Publication No. 2001/0036670 issued to Fryer et al. (“Fryer”). Applicants respectfully traverse the rejections.

To establish a *prima facie* case of obviousness, the Examiner must set forth “some articulated reasoning with some rational underpinning to support the conclusion of obviousness.” See KSR International Co. v. Teleflex Inc., 82 USPQ2d 1385, 1396 (2007). In combining prior art elements to render the claimed combination of elements obvious, the Examiner must show that the results would have been predictable to one of ordinary skill in the art. See Examination Guidelines for Determining Obviousness Under 35 U.S.C. 103, Section III(D), issued by the U.S. Patent and Trademark Office on October 10, 2007.

In regard to independent claim 1, Applicants respectfully submit that Omatsu and Fryer fail to disclose or render predictable at least the elements of “the light-transmitting gas selective permeable film comprising a thickness of 0.05 μm to 1 μm” as recited in amended claim 1.

Omatsu discloses an ozone indicator and concentration measuring method. The Examiner alleges that Omatsu discloses a changing layer (“porous material”), an ink containing anthraquinone dye (“sensing agent”) and an overcoat (“gas selective permeable film”). The Examiner admits Omatsu is silent regarding the selective permeability of the permeable film and instead alleges Fryer discloses that it is desirable to provide a layer over the sensing layer that is gas permeable. See Action, page 2.

Nowhere, however, within Omatsu or Fryer is a thickness of the alleged light-transmitting gas selective permeable film disclosed. Accordingly, the cited prior art references fail to disclose a light-transmitting gas selective permeable film comprising a thickness of 0.05 μm to 1 μm as required by amended claim 1.

For at least the foregoing reasons, Omatsu and Fryer fail to disclose or render predictable each and every element of claim 1. Since each of the elements of claim 1 is not found within the cited prior art references, a *prima facie* case of obviousness may not be established. Applicants respectfully request reconsideration and withdrawal of the rejection of claim 1 under 35 U.S.C. §103 in view of Omatsu and Fryer.

Claims 2, 3, 5 and 13-15 depend from claim 1 and incorporate the limitations thereof. Thus, for at least the reasons that claim 1 is not *prima facie* obvious over Omatsu and Fryer, claims 2, 3, 5 and 13-15 are further not obvious over the cited prior art references. Applicants respectfully request reconsideration and withdrawal of the rejection of claims 2, 3, 5 and 13-15 under 35 U.S.C. §103 in view of Omatsu and Fryer.

**B.** In the outstanding Action, claims 4, 6-12 and 17 are rejected under 35 U.S.C. §103(a) as being unpatentable over Omatsu in view of Fryer and further in view of U.S. Patent Publication No. 2004/0131501 issued to Maruo (“Maruo”). Applicants respectfully traverse the rejections.

Claims 4, 6-12 and 17 depend from claim 1 and incorporate the limitations thereof. For at least the reasons previously discussed, Omatsu and Fryer fail to disclose or render predictable at least the elements of “the light-transmitting gas selective permeable film comprising a thickness of 0.05  $\mu$ m to 1  $\mu$ m” as incorporated into claims 4, 6-12 and 17 from claim 1. The Examiner has further not pointed to, and Applicants are unable to discern, a portion of Maruo curing the deficiencies of Omatsu and Fryer with respect to these elements. Thus, for at least the foregoing reasons, claims 4, 6-12 and 17 are not *prima facie* obvious over Omatsu, Fryer and Maruo. Applicants respectfully request reconsideration and withdrawal of the rejection of claims 4, 6-12 and 17 under 35 U.S.C. §103 in view of Omatsu, Fryer and Maruo.

C. In the outstanding Action, claims 18-36 are rejected under 35 U.S.C. §103(a) as being unpatentable over Omatsu in view of Maruo. Applicants respectfully traverse the rejections.

In regard to independent claim 18, Applicants respectfully submit that Omatsu and Maruo fail to disclose or render predictable at least the elements of “an ozone sensing sheet formed by carrying a dye containing an indigo ring and a humectant by a sheet-like carrier made of fibers, wherein said humectant is operable to accelerate a reaction between the dye and ozone” as recited in claim 18.

As previously discussed, the Examiner alleges that Omatsu discloses an ozone indicator and concentration measuring method. The Examiner alleges that Omatsu discloses a changing layer (“porous material”), an ink containing anthraquinone dye (“sensing agent”) and an overcoat (“gas selective permeable film”). The Examiner alleges that Maruo discloses an ozone gas sensing element comprising a dye solution prepared by dissolving an indigo carmine disodium salt, acetic acid and glycerol. Apparently recognizing the failure of the references to disclose the element of “wherein said humectant is operable to accelerate a reaction between the dye and ozone,” the Examiner alleges this element is an inherent property since the claimed humectant is disclosed by Maruo. Applicants respectfully disagree.

In particular, although Maruo teaches to add a humectant to an arrangement carrying a dye in a transparent porous material, Maruo does not teach or contemplate use of a humectant in combination with an arrangement in which a dye is carried by a sheet-like carrier made of fiber.

Applicants respectfully submit that a humectant becomes active to accelerate the reaction between the dye and ozone only if the humectant is used in combination with an arrangement in which the dye is carried by a sheet-like carrier made of fiber as claimed. As evidence of this fact, Applicants respectfully submit for the Examiner's reference, an article by the present inventors, Maruo et al., entitled *Development and evolution of ozone detection paper*, Sensors and Actuators B 135 (2009) 575-580. On page 576, left column, lines 5-7, of the article, it is disclosed that the addition of a humectant to an arrangement carrying a dye in a transparent porous material is not effective to accelerate a reaction between a dye and ozone. Accordingly, a humectant does not naturally have an action or effect to accelerate a reaction between a dye and ozone. This is a novel action unknown in the prior art that will be developed or exhibited only if a humectant is applied to the claimed arrangement in which a dye is carried by a sheet-like carrier made of fiber.

Since the alleged humectant in Maruo is not carried by the claimed sheet-like carrier made of fiber, it would not inherently accelerate a reaction between the dye and ozone disclosed by the prior art as alleged by the Examiner. Accordingly, the combination of Omatsu and Maruo fails to explicitly or inherently disclose that when a dye is carried by a sheet-like carrier made of fiber, a humectant which is carried by the carrier together with the dye acts to accelerate a reaction between the dye and ozone as required by claim 18.

For at least the foregoing reasons, Omatsu and Maruo fail to disclose or render predictable each and every element of claim 18. Since each of the elements of claim 18 is not found within the cited prior art references, a *prima facie* case of obviousness may not be established. Applicants respectfully request reconsideration and withdrawal of the rejection of claim 18 under 35 U.S.C. §103 in view of Omatsu and Maruo.

Claims 19-36 depend from claim 18 and incorporate the limitations thereof. Thus, for at least the reasons that claim 18 is not *prima facie* obvious over Omatsu and Maruo, claims 19-36 are further not obvious over the cited prior art references. Applicants respectfully request reconsideration and withdrawal of the rejection of claims 19-36 under 35 U.S.C. §103 in view of Omatsu and Maruo.

**CONCLUSION**

In view of the foregoing, it is believed that all claims now pending, namely claims 1-36, are now in condition for allowance and such action is earnestly solicited at the earliest possible date. If there are any additional fees due in connection with the filing of this response, please charge those fees to our Deposit Account No. 02-2666. Questions regarding this matter should be directed to the undersigned at (310) 207-3800.

Respectfully submitted,

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**CERTIFICATE OF TRANSMISSION**

I hereby certify that this correspondence is being submitted electronically via EFS Web to the United States Patent and Trademark Office on August 24, 2009.

  
Si Vuong



## Development and evaluation of ozone detection paper

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### ABSTRACT

A high sensitive paper with good response has been developed for ambient ozone monitoring. The paper operates by employing the reaction of ozone with indigo carmine. This reaction results in white-yellowish reaction products on the paper that can be determined by measuring the paper's reflectance. Direct, on-site measurements are possible because of the selectivity of the color change reaction, and only a small photoelectric meter is needed. The concentration of ozone, shear, humectant, acid and indigo carmine is needed to achieve a good response and the color of our developed ozone detection paper fades completely from blue to white when exposed to ozone concentrations of about  $400 \text{ ppb} \times \Delta t$ . The humectant content strongly affects the ozone response, and a 30% glycerol content of the paper in mixed solution yields the best response. We also measured the effects of temperature, relative humidity and wind speed on the ozone response, and found that relative humidity has a strong influence on the ozone response. The interference gases were checked for six chemicals that are usually present in the air, and only hydrogen peroxide exhibited positive interference. Since our developed sensing paper has the good response, it is suitable for hourly to daily ozone measurements of ambient ozone and we successfully measured indoor ozone concentrations using our developed ozone detection paper and a photoelectric meter with a single wavelength source.

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### 1. Introduction

Ozone is an atmospheric oxidant formed by photochemical reactions between volatile organic compounds (VOCs) and nitrogen oxides in the presence of sunlight [1]. Although a great deal of effort has been aimed at reducing the emissions of these ozone precursors, ambient ozone concentrations have not decreased over the last decade [2–7]. At the ambient concentrations, ozone exposure can cause respiratory/health effects, including changes in lung capacity, flow resistance, and epithelial permeability [8]. Ozone is also emitted from germicide lamps, copy machines, printers, during welding and other industrial processes. Thus, the investigation of human health effects associated with ozone exposure requires knowledge of both outdoor and indoor concentrations as well as personal exposure measurements [9,10]. Although a great deal of information is available about outdoor ozone concentrations and personal exposure [11,12], very little is known about indoor concentrations and personal exposure [13,14]. One reason for this is the lack of lightweight, inexpensive, and reliable ozone monitors suitable for indoor or personal monitoring. Various ozone detection methods have been reported, including spectroscopy, chemiluminescence

and chemical techniques. The passive sampler method is simple, inexpensive, does not require a pump, and is widely used for exposure points [15–18]. Moreover, it offers a flexibility that allows us to realize more ambitious sampling schemes, which can include both indoor and personal monitoring. The time resolution of the passive sampler technique is limited, as it can only provide information on integrated average concentrations over the exposure period (weekly in many cases). Sodium nitrite ( $\text{NaNO}_2$ ) is promising trapping reagent in detecting ambient ozone because it offers sufficient sensitivity, few interference problems [19,20]. Less favorable features of the method are the time consuming analysis and poorer time resolution. Indigo carmine seems to have the best ozone selectivity of a number of potential compounds such as curcumin. The drawback of this method is that low sensitivity, poor reproducibility and positive interference of nitrogen dioxide [21]. An indigo-coated fiber with high sensitivity has been reported [22], however the drawbacks of this method are the interference of  $\text{NO}_2$  up to 30%, the need for chemical analysis and the need to use a pump for sampling.

The aim of this work is to develop a passive accumulated sensing paper for ozone monitoring that offers good response and high-time resolution but without the need for chemical treatment such as dissolution or the use of a pump for sampling. We have already reported a passive accumulated ozone sensor based on the above principle [23], and it can detect hourly changes in ozone concentration as effectively as a large analytical instrument using a sensing

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device with LEDs, PDS, and a passive accumulated sensor element. However, the sensitivity of the ozone porous glass-sensing element is slightly low and it requires more than a week (accumulated ozone concentration: 17,000 ppb  $\times$  h) for the blue color to fade completely. A humectant did not work well in terms of obtaining a highly sensitive ozone-sensing element when we used porous glass as a substrate. However, a humectant provided the good response when we used a cellulose sheet as a substrate. We then fabricated a simple ozone detection paper with a humectant on the cellulose sheet. This paper could effectively detect hourly changes in ozone concentration, and its color faded completely from blue to white when exposed to concentrations of about 400 ppb  $\times$  h. We also found that the paper was 30 times more sensitive than a glass sensor.

## 2. Experimental

## 2.1. Sensor network

For the sensor substrate we used 0.26 mm thick cellulose sheets (qualitative filter paper no. 2; Advantec Toyo, Japan). The sheets had an average free volume of 78%, a retention particle diameter of 5  $\mu$ m and a density of 125 g/m<sup>2</sup>, and they were cut into 20 mm  $\times$  20 mm squares.

Commercial grade indigo carmine, glycerol, ethylene glycol, propylene glycol, citric acid, lactic acid, propionic acid and acetic acid were purchased from Kanto Chemical Co. (Japan), and were used without further purification.

We prepared an aqueous mixture solution of 50 g containing 0.045 g of Indigo carmine, 3.5 g of citric acid and 12.5 g of glycerol. The cellulose sheets were immersed in a container filled with the solution including the reagents for 30 s, and then dried in dry nitrogen gas for 1 day to remove the water. The ozone detection papers fabricated in this method were used for all our experiments except for the experiment on the effect of humectant content, humectant species and acid species.

For our experiment on the effect of humectant content and humectant species on the degree of color fading, we used three kinds of humectant (glycerol, ethylene glycol, and propylene glycol) and prepared 21 kinds of 50 g aqueous solution containing 0.045 g of indigo carmine, 3 g of acetic acid and various amounts of humectant.

For our experiment on the effect of acid species on the degree of color fading, we used four kinds of acid (citric acid, lactic acid, propionic acid and acetic acid) and prepared 23 kinds of 50 g aqueous solution containing 0.045 g of indigo carmine, an acid and various amounts of dissolved

### 2.3. Gas separation and measurement

Gas mixtures containing between 30 and 150 ppb levels of ozone were prepared in a DKK ozone generator (Tos-DKK, Japan) where ozone was generated by the electric discharge of compressed air. The samples were exposed to the gas by placing them in a temperature and humidity controlled chamber (EquiDrier, Iwate Co. Ltd., Japan) and allowing the gas to flow over them. The wind speed in the chamber was about 1.5 m/s.

To estimate the effect of wind speed, a centrifugal blower was installed at the inner side of the chamber, and the wind speed was adjusted by controlling the blower voltage. The wind speed was measured with an anemometer (KANOMAX, Japan).

For NO or NO<sub>2</sub> exposure experiments, air containing artificial NO or NO<sub>2</sub> gas flowed into the chamber at 3 l/min. The exotoxins detection chamber was placed on a plastic plate, and the plate was then placed

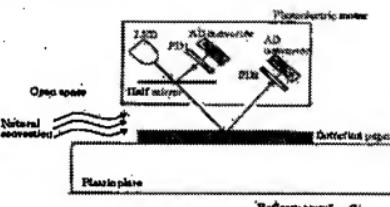


Fig. 1. Experimental set up for measuring reflection values at 611 nm using developed photoelectric meter.

in the center of the chamber at 25°C, and 40% RH. The NO and NO<sub>2</sub> concentrations were measured using a Japan Thermo (JTE) 42C analyzer. The NO or NO<sub>2</sub> concentration was about 100 ppb.

For exposure experiments with other interference gases (hydrochloric acid or formaldehyde or acetdehyde or hydrogen peroxide), we placed a transpiration source in a Tedlar bag filled with ambient air [24] at 20 °C, and 905 RH. The transpiration source was a borosil glass tube filled with diluted liquid formalin, diluted acetdehyde aqueous solution, diluted hydrochloric acid solution or diluted hydrogen peroxide solution, all of which were obtained from Kanto Chemical Co. Inc. (Japan). The opening of the bottle was covered with an 80  $\mu$ m thick Teflon membrane filter (Advantec Toyo (Japan); TD20A) with a pore size of 0.2  $\mu$ m. The gas concentration was checked with gas detection tubes (GASTECH (Japan)) of 14L for hydrochloric acid, 32L for hydrogen peroxide, 911L for formaldehyde, and 92L for acetdehyde, respectively.

In the ambient ozone measurement set up, the samples were placed on a plastic plate and this was placed on the table in the experimental room. The temperature and relative humidity in the room were measured simultaneously using sensors (SH1175, Sensirion Swiss). The experimental room was constantly ventilated; therefore the ozone concentration in the experimental room was almost the same as that outside. The ozone concentrations were measured using a Japan Thermo (ITB) 450C analyzer.

The reflection spectra of the samples were measured using a Hitachi U-3500 spectrometer with a reflection measurement unit. A mirror was used as reference. The reflection spectra of the ozone detection paper ranged from 200 to 800 nm, and the reflectance represented the ratio between the output intensities of the sample and the reference.

## 2. Results and discussion

### 3.1 Reaction between ozone and ozone detection paper

We examined the color fading reaction that occurred when ozone came into contact with our ozone detection paper by exposing the papers to a ozone gas flow of about 80-90 ppb for 0-8 h at 20°C and 60% RH. After the exposure, the blue paper had turned into a pale blue. Fig. 2 shows the change in the reflectance spectra of the paper. A single reflection peak appeared at 518 nm, and it was clear that the common logarithm of (1/reflectance) decreased as the accumulated ozone concentration increased.

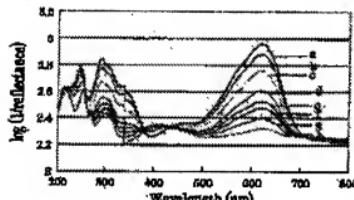


Fig. 2. Changes in reflectance spectra of developed ozone detection paper after exposure to 0-416 ppb  $\times$  h, at 25°C, and 60% RH: (a) 0 ppb  $\times$  h, (b) 64 ppb  $\times$  h, (c) 128 ppb  $\times$  h, (d) 192 ppb  $\times$  h, (e) 256 ppb  $\times$  h, (f) 287 ppb  $\times$  h, (g) 350 ppb  $\times$  h, and (h) 416 ppb  $\times$  h.

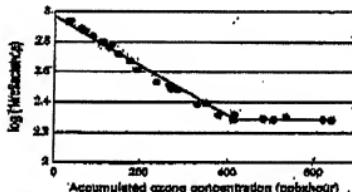


Fig. 3. Relationship between  $\log(1/\text{reflectance})$  at 618 nm and exposure accumulated ozone concentrations. Twenty-eight samples were used in this experiment and the exposure conditions were 25°C, and 60% RH.

We focused on the reflectance change at 618 nm. Fig. 3 shows the relationship between the accumulated exposed ozone concentration and the  $\log(1/\text{reflectance})$  change at 618 nm for 28 sheets of ozone detection paper exposed to different accumulated ozone concentrations at 25°C, and 60% RH. The  $\log(1/\text{reflectance})$  decreases with increasing exposure up to an accumulated ozone concentration of about 400 ppb  $\times$  h, and remained constant at more than 400 ppb  $\times$  h. The constant value means that the color has completely faded. The detection paper is white in this flat region and the value of  $\log(1/\text{reflectance})$  is constant at 2.30. A good linear relationship was obtained between the  $\log(1/\text{reflectance})$  and the accumulated ozone concentration up to about 400 ppb  $\times$  h. This relationship is shown by Eq. (1):

$$O_3 (\text{ppb} \cdot \text{h}) = -570 \times \log \left( \frac{1}{\text{reflectance}} \right) + 1700 \quad (1)$$

In this equation,  $O_3$  (ppb  $\times$  h) indicates the accumulated exposed ozone concentration.

Although these plots included several experimental conditions with different ozone exposure concentrations of 30-150 ppb, all the data were on the same line when we used the accumulated ozone concentration as the X-axis. This shows that the sensing paper worked as accumulated one.

Fig. 4 is a photograph of a series of ozone detection papers exposed to different accumulated ozone concentrations at 25°C and 60% RH. As shown in Fig. 4, the blue ozone detection paper turned completely white after exposure to an ozone level of 416 ppb  $\times$  h.

### 3.2. Precision of developed ozone detection paper

To estimate the paper's precision, we used nine samples for one experiment. The ozone exposure was performed at a temperature of 20°C, and 60% RH. The total ozone concentration to which the samples were exposed was 380 ppb  $\times$  h. The variation of the difference in the  $\log(1/\text{reflectance})$  at 618 nm across the samples was within  $\pm 10\%$  of the mean value.

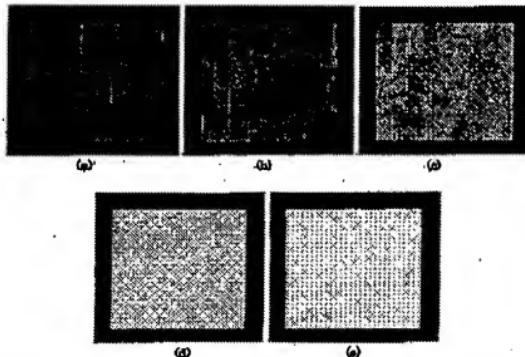


Fig. 4. Photographs of developed ozone detection paper after exposure to 0-640 ppb  $\times$  h ozone at 25°C, and 60% RH: (a) 0 ppb  $\times$  h, (b) 128 ppb  $\times$  h, (c) 256 ppb  $\times$  h, (d) 416 ppb  $\times$  h, and (e) 640 ppb  $\times$  h.

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## Biographies

Yasuhiko Yamaaji Mares received a BSc degree in 1994, an MSc in 1996, and a PhD in 2003 from Tohoku University, Sendai. She is currently engaged in research on chemical sensors for atmospheric environment at NTT Energy and Environment Systems Laboratories.

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Reo Nakamura received BE, ME, and PhD degrees in applied chemistry from Osaka University, Osaka, in 1987, 1989, and 1995, respectively. He is currently engaged in the development of environmental-sensing systems and analysis of the influence of  $\text{NO}_x$  on the global environment at NTT Energy and Environment Systems Laboratories. He received the MicroProcess Conference Award in 1997 and the Photopolymer Conference Award in 1998.